Name: _____

Lab Instructor:_____

PREPARATION FOR CHEMISTRY LAB: A CHEMICAL SYNTHESIS

1. Draw the structural formulas (similar to Lewis Structures, see the reaction in the introduction) for salicylic acid and acetic acid.

2. What is a catalyst (if necessary, use your glossary)? What is the catalyst used in this week's lab?

3. What is the common name for acetylsalicylic acid?

4. Transfer these results to the report sheet.

   a) What is the molar mass of salicylic acid?

   b) What is the molar mass of acetic anhydride?

   c) What is the molar mass of acetylsalicylic acid?

5. 5.68 grams of salicylic acid and 4.77 mL of acetic anhydride (density: 1.080 g/mL) are mixed together. 3.90 grams of acetylsalicylic acid are obtained in the reaction.

   What is the theoretical yield of the reaction?

   What is the percent yield of the reaction?

A CHEMICAL SYNTHESIS

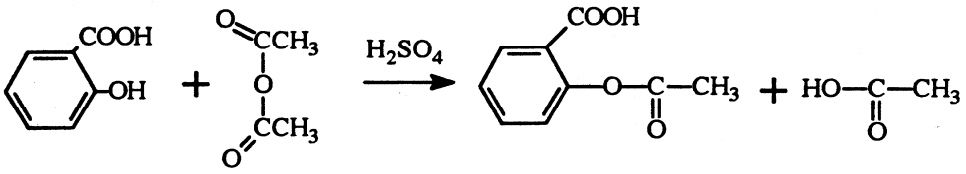
Chemical synthesis is the laboratory preparation of a chemical compound. New compounds, like medicines, can be tailor-made for specific uses. Natural compounds can be synthesized from cheaper raw materials. For example, vanillin (vanilla flavor) is made in the laboratory using waste from the wood pulp industry for far less than it costs to extract the compound from vanilla beans.

INTRODUCTION

So far our labs have mainly dealt with measurement and chemical analysis. However, in this lab we will be synthesizing a chemical compound, acetylsalicylic acid (aspirin), from salicylic acid and acetic anhydride as shown in the equation below. Sulfuric acid is a *catalyst* for this reaction. We will use a process called vacuum filtration to obtain crystals of product from the reaction mixture and measure the melting point (mp) of the compound as a test of its purity. The presence of impurities lowers the mp of a pure compound.

Read and/or review Sections 7-1 through 7-3 in your textbook.

Reaction:



salicylic acid acetic anhydride aspirin (acetylsalicylic acid) acetic acid

Each of the ring structures shown above has 6 carbon atoms (one at each point of the hexagon) and 4 hydrogen atoms (bonded to the 4 carbon atoms in the ring structure that don't already show something bonded to them). Thus, the formula for salicylic acid is $C_7H_6O_3$ and the formula for acetylsalicylic acid is $C_9H_8O_4$.

PROCEDURE:

PREPARATION:

Set up a boiling water bath using DI water at your work station. Place 25 mL of DI water in a large test tube (TT_1) and place it in the water bath. Place a plastic squirt bottle containing water on ice.

Weigh 1 g of salicylic acid and transfer it to a large, dry test tube (TT_2). A preset pump dispenser is set up in the **hood.** It contains the **flammable and corrosive** liquid, acetic anhydride (density: 1.080 g/mL). Pump 2 mL into TT_2. **Slowly and carefully** add 3 drops of concentrated sulfuric acid from a dropper bottle. Place TT_2 in your boiling water bath and continue heating for 5 min after the solid has dissolved.

Pour the 25 mL of boiling water from TT_1 into a 100 mL beaker. Add the contents of TT_2 to the beaker. Allow the beaker to cool to room temperature. After the beaker has reached room temperature, cool the beaker on ice and scratch the bottom of the beaker with a stirring rod. These actions initiate crystallization though the process may take several minutes.

While cooling is underway, examine the vacuum filtration unit set up in the lab near your work station. Your lab instructor will give additional hints on technique. With the vacuum pump running and the filter paper firmly seated in the funnel, quickly pour the cooled product in your beaker onto the center of the filter paper.

Rinse any remaining product in the beaker onto the filter using a few mL of ice-cold water from the squirt bottle, washing the product on the filter with the rinse. Repeat the rinse and wash twice more.

Spread out the ppt on the filter paper and allow the pump to run a few minutes longer until the product is fairly dry. After the product has dried, transfer it to a tared small, clean, dry beaker and determine the mass of product. The solid obtained in this step is impure aspirin. Prepare two capillary sample tubes using this impure product to use in the melting point portion of the lab in case you do not get enough of the purified aspirin to use. Purify the remaining impure aspirin according to the following procedure.

PURIFICATION BY RECRYSTALLIZATION:

Obtain a plastic pipet and flush it with boiling water from your boiling water bath.

Very slowly add hot water from the water bath using the plastic pitpe, a few drops at a time, until the product just dissolves. Allow the beaker to cool to room temperature. Scratch the bottom of the beaker with a stirring rod if crystals have not formed. Once crystals have started to form, cool the beaker on an ice bath. Be patient, slow cooling favors the formation of crystals.

Vacuum-filter the crystals and wash them with **ice water**. Use the plastic squirt bottle containing ice-cold water to squirt a small amount of ice water over the crystals. Again, allow the pump to run for a few minutes until the crystals are dry. Carefully remove the paper and product from the filter and lay them on a watch glass. Set the watch glass on a hot plate (very low heat) for about 10 min.

After the product has cooled, transfer it to a tared container and determine the mass of purified aspirin.

DETERMINATION OF THE MELTING POINT OF THE PRODUCT:

Your lab instructor will demonstrate the use of the electrically-heated melting point units in the lab. Prepare two capillary sample tubes of the purified aspirin (use the tubes containing impure aspirin you prepared earlier if you did not get purified aspirin) and obtain the average melting point for your product.

DATA AND ANALYSIS SHEET: A CHEMICAL SYNTHESIS

Name: _____

DATE_____ LAB PARTNER_____

Molar mass of salicylic acid: _____

Mass of salicylic acid used: _____

Moles of salicylic acid used: _____

Molar mass of acetic anhydride: _____

moles of acetic anhydride (density: 1.080 g/mL) used: _____

Molar mass of acetylsalicylic acid: _____

Mass of impure aspirin obtained: _____

Mass of purified (recrystallized) aspirin obtained: _____

Calculate the theoretical yield, in grams, for the reaction using 2.00 mL of acetic anhydride and **your** starting amount of salicylic acid.

Determine the percent yield for the reaction using your experimental results for the mass of the purified aspirin obtained (use the mass of impure if you did not get any purified aspirin).

Explain/justify your percent yield results.

Melting points of the two purified aspirin samples: _____ ; _____

Melting points of the two impure aspirin samples if you used impure instead: _____ ; _____

Average melting point:_____

Melting point given in the literature: _____

Explain/justify your melting point results.

Name: _____

Lab Instructor:_____

QUESTIONS ABOUT THIS LAB: A CHEMICAL SYNTHESIS

1. How many moles of salicylic acid did you use in this synthesis? How many moles of acetic anhydride did you use? Theoretically, how many moles of aspirin should you get?

2. A commercial aspirin tablet contains 325 mg of aspirin. How many tablets could you make from your final product?

3. Assuming that a kilogram of pure acetylsalicylic acid sells for $54.50. What would be the cost of a single commercial aspirin tablet that contains 325 mg of aspirin?

4. Aspirin is an acid. Which analytical method used this semester would be the most suitable for the measurement of the amount of aspirin in an impure aspirin sample? What chemicals would you need? Briefly discuss the procedure you would use.

5. 5.04 grams of salicylic acid and 2.18 mL of acetic anhydride (density: 1.080 g/mL) are mixed together. 2.43 grams of acetylsalicylic acid are obtained in the reaction. What is the percent yield of the reaction?